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(54) IMPROVEMENTS IN AND RELATING TO STERILISATION

(71) We, TETRA PAK INTERNATIONAL, A.B., a Swedish corporate body of Fack S-221 01, Lund 1, Sweden, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the surface sterilisation of an article or material, and is *inter alia* suitable for sterilising packaging material, and blanks for packages.

Sterile packages of the disposable type are known and are used, *inter alia*, for packing liquid foods, e.g. milk. The contents packed in sterile packages have an appreciably longer keeping period than contents packed in non-sterile packages. The known sterile packages used, for example, for liquid dairy products, are of various types. Common to them all is, however, the procedure required for the manufacture of sterile packages. Both the intended contents and the packaging material, or the package blanks partly formed from packaging material, are sterilized, and the contents are filled in, and the packages are closed, under aseptic conditions.

A well-known consumer package for beverages, e.g. milk, is manufactured from packaging material, which may consist, for example, of a laminate comprising a carrier layer of fibrous material covered on each face with a thin plastics layer. This is fed into a packaging machine in the form of a web. During the passage of the web through the machine it is folded to tubular shape, by bringing its opposite longitudinal edges into overlapping positions and sealing them together. This formation of the web into a tube takes place continuously during the advance of the web, in a generally vertical path downwards through the machine. After the formation of the web to tube-shape, the contents are fed into the tube continuously through a filler pipe which extends into the tube through the upper open end. During the downward advance of the loaded tube it is pressed flat and sealed by sealing jaws located on opposite sides of the tube to seal the tube along narrow transverse zones spaced from one another. The supply of contents is continuously controlled automatically in such a manner that the level of contents is well above the point where the tube is pressed flat and sealed. After the sealings, the tube is cut transversely through the sealed zones, and, after reshaping of the individual packages if desired, the manufacturing process is complete.

When a machine such as described above is used for producing sterile packages, the filling takes place under aseptic conditions, which means that the atmosphere in the material tube, as well as the material of the tube itself (or in any case its inside surface) must be kept sterile. For the former purpose a prearranged super-pressure of sterile air is maintained in the tube so that non-sterile air cannot penetrate from the surrounding atmosphere.

The sterilization of the web material is effected in a known machine of the kind described, by passing the web, before forming into a tube, through a bath of chemical sterilizing agent, usually a solution of hydrogen peroxide, which moistens the material, whereafter the excess liquid is removed from the web by pressure rollers. The sterilizing agent which remains on the web is, after the formation of the web to a tube, removed from the inside of the tube by heating means, usually a heating coil arranged around the filler pipe, (a so-called tube heater), which heats the inside of the tube so that the residue of sterilizing agent remaining thereon evaporates and escapes from the upper, open end of the tube.

This method of sterilization has been found to be subject to certain disadvantages. Thus it has been found, *inter alia*, to be difficult under certain conditions to control the quantity of sterilizing agent on the web with sufficient accuracy, since the pressure rollers used for the purpose render it difficult to meter the result accurately. To ensure a uniform application of sterilizing agent on the web, and hence an effective sterilization, the sterilizing agent should contain a stabilizer and wetting agents, which substances are difficult to remove completely, and which

are moreover undesirable for reasons of cost.

Another method for sterilizing the packaging web in the type of machine described has been proposed in co-pending Patent Application No. 47925/76 (Serial No. 1.561,495), in which a liquid sterilizing agent, preferably a mixture of hydrogen peroxide and water is introduced into a dish-like container located inside the tube. This container surrounds the filler pipe and is heated to such a temperature that the sterilizing agent, which is delivered drop-wise to the dish, evaporates immediately. The vapor rises upwards through the tube and is deposited at the upper end of the inside wall of the tube. During the continuous downward movement of the tube, the vapour condensed at a certain moment at the upper end of the tube passes the heating element located lower down in the tube, and this heats the inside wall of the tube as well as the condensate thereon so vigorously that the agent is evaporated and rises upwards to the regions of the upper end of the tube where it is recondensed on the colder tube wall. This process is repeated as long as the machine is in operation, and the whole time a dropwise delivery of the sterilizing agent to the heated dish takes place in order to replace the loss which arises through part of the evaporated sterilizing agent rising upwards and escaping through the upper end of the tube without being condensed on the tube wall.

A disadvantage of the latter method is that the sterilizing agent used is mixed with a fairly large proportion of water, and the actual concentration of sterilizing agent in the mixture is only in the range of 10—35% by weight. The vapour produced thus consists largely of water vapor which is undesirable for sterilization, since it reduces the bactericidal effect. It is also difficult to control the process with sufficient accuracy, since it is essential that the quantity of condensed sterilizing agent is not too great to be completely evaporated when the tube passes the heating element, since otherwise sterilizing agent may remain in the packages.

In a further known type of machine for packaging liquid contents, the packages are not manufactured from a web of material, but from prefabricated blanks provided with an outer contour and a creaseline-pattern, which facilitates the forming of the finished containers. The blanks are folded to form flattened tubular bodies which are stacked into the magazine of the packaging machine. From here the tubular bodies are introduced one-by-one into the machine to be opened into a tube, and by folding in of flaps at one end, to be formed with a base. The containers, open at the top, are deposited subsequently on a belt by which they are conveyed past a number of processing stations, namely a station f r the application of sterilizing liquid to the inside of the containers, a station for the removal of the sterilizing liquid, a station for the introduction of the desired contents into each container and a station for the sealing of the upper end of the container. The station for the sterilization of the containers comprises a spray nozzle which is located above the container and, so to speak, "spray paints" the inside of each container with sterilizing liquid. The sterilizing liquid acts during the time the container is moved to the next station at which hot air is blown down into the container over such a period that the sterilizing liquid evaporates completely and escapes from the upper open end of the container.

This method of sterilization is subject first and foremost to two disadvantages, namely certain recesses and corners at the bottom end of the container cannot easily be reached by the sterilizing liquid, and that a relatively large quantity of sterilizing liquid has to be used, so that the excess is liable to accumulate at the base of the container where residues may possibly remain.

An object of the invention is to provide a method of, and means for, sterilization which will be free from the disadvantages of known procedures mentioned above, and with this end in view the invention consists in a method of sterilizing the surface of an article or material wherein the surface at a relatively low temperature is exposed in a substantially closed chamber to an atmosphere comprising a vaporised liquid sterilising agent and a gas mixed in another chamber remote from, and conducted and delivered into, said first-mentioned chamber under conditions which ensure condensation of some of said vapour on said surface, and, after a pre-arranged period of time, said condensed sterilising agent is removed from said surface.

By a relatively low temperature is meant a temperature low enough to ensure said condensation.

The invention also consists in apparatus adapted to carry into effect the aforesaid method comprising a chamber for preparing a mixture of vaporised liquid sterilizing agent and a gas, means for conducting and delivering said mixture to another chamber housing an article or material for sterilization under conditions which ensure condensation of same of said vapour on the surface thereof, and means for subsequently removing said condensate sterilizing agent from said surface and conducting it from said second-mentioned chamber.

A preferred form of the invention will now be described by way of example with reference to the accompanying drawing which shows schematically means for carrying into effect the method in accordance

with the invention in a packaging machine of the type which produces packages from a continuously moving web.

The principle of the present invention will first be explained to facilitate understanding thereof. The capacity of air to absorb, carry and give off moisture is a function of the temperature. The capacity increases with increasing temperature and decreases with decreasing temperature. If a given amount of atomized liquid or vapour is injected into heated air, it is absorbed and carried by the air as long as the temperature is sufficiently high, that is to say higher than the condensation temperature or dew-point of the mixture. When for whatever reason the temperature of the air thus moistened drops below the condensation temperature or dew-point, it can no longer carry such a large amount of liquid, and part of the liquid is precipitated. This can be made use of for coating a surface with a uniform layer of the liquid vapour carried by the air, which takes place if a body of a lower temperature than the condensation temperature of air and vapour mixture is introduced into it. That part of the air which comes into contact with the body is cooled thereby and the moisture is precipitated and condenses on the surface of the body. During the condensation, heat is liberated which warms the cooler body and the condensation continues therefore only as long as the surface is of a sufficiently low temperature. The quantity of condensed liquid is a direct function of the relative temperature of the surface, the specific heat and the thermal conductivity of the body. If these factors are identical for the whole surface, the process is exactly the same on each surface area and in this manner a completely identical condensation can be ensured over the whole surface which is thus coated with a uniform layer of the condensed liquid.

The abovementioned principle is in accordance with the invention, employed to coat a surface, for the purpose of sterilization with a uniform layer of a liquid sterilizing agent. Since air normally contains some water vapour it is desirable, in order to avoid dilution of the sterilizing agent, initially to remove as much water as possible from the air, i.e. the air should be as dry as possible. This dry air is heated so that it can evaporate the sterilizing agent and thereafter remain at a sufficient temperature for carrying the desired quantity of sterilizing agent. By controlling the temperature of the air, and the ratio between the quantity of sterilizing agent and the quantity of air, the condensation temperature or dew-point of the mixture of air and vapour of the sterilizing agent can be controlled to a value which by a prearranged amount exceeds the temperature of th surface which is to be coated with sterilizing agent. When the desired mixture of air and vapour has been obtained, the mixture is applied to the surface which is to be sterilized, whereby the air is cooled and its capacity for carrying sterilizing agent is diminished so that a large portion of the sterilizing agent condenses on the surface. After a suitable reaction time, which will depend on the type of sterilizing agent and its concentration, as well as on the bactericidal effect required, the sterilizing agent is removed again. This may be done in any of various ways, e.g. by hot air or by irradiation with any heating element, e.g. an infra red lamp.

The method in accordance with the invention will now be described in more dtail, such as it may be used in a packaging machine of known type. In the accompanying schematic drawing the machine is shown on the right-hand side of the drawing. The machine operates with a web of material 1, drawn from a roll 2, which web passes through two chambers 3, 4 in sequence. The web 1 is passed over two guide rollers 5 and substantially vertically downwards through the packaging machine. During the downward movement through the machine the web is transformed into a tube 6 by bonding its two longitudinal edges together to form a longitudinal seam. After formation and sealing of the web to tubular form the tube is filled with the required contents, e.g. milk, through a filler pipe 7 which extends through the upper, open, end of the tube. At a distance below the lower end of the filler pipe are located the sealing jaws 8, which, at equal intervals in the filled tube 6 as it moves vertically downwards, provide transverse seals through which the tube is transversely divided. The completely filled packages 9 so formed are conveyed to a processing or final folding station 10 where they are given a parallelepipedic form.

The sterilizing system of the invention which co-operates with the machine comprises as air compressor 11, a cooler 12 for the compressed air connected to the compressor, and a water separator 13 wherein the moisture precipitated from the air is removed. The dehydrated air is conducted by a pipe system to two regulating valves 15, 16 which control the steady flow. The bulk of the air is passed through the regulating valve 15 to a heating arrangement or an element 17 which heats the air to the desired extent. From the heating element 17 the air flows into a chamber 18. At the upper end of this chamber is located an ultrasonic nozzle 19 which is operated by the part of the air stream from the water separator 13 controlled by the regulating valve 16. To the ultrasonic nozzle 19 also leads a supply line for sterilizing agent which is fed from a tank 20 through suitable lines and a pump 21 to the ultrasonic nozzle 19. The ultrasonic

nozzle is of a known design and functions in such a manner that the air stream from the regulating valve 16 creates a standing wave of a very high frequency (approximately 30 kHz). Into this standing wave is introduced sterilizing agent which is atomized to droplets of a size 5—15 micrometre. This highly atomized liquid or mist is mixed in the container or mixing chamber 18 with the hot air whereby the mist changes into vapour which through a pipe 22 is passed to the previously mentioned chamber 3, through which the web material passes on its way from the roll 2 to the tube-forming part of the machine. In the chamber 3 the mixture of sterilizing agent and air comes into contact with the moving web 1 which cools the mixture so that the sterilizing agent condenses to a uniform layer on the web surface. Part of the sterilizing agent condenses on the wall of the chamber and is recovered through a drain pipe 23.

As mentioned previously, the web 1 passes through another chamber 4 which, in the direction of movement of the web, is spaced from the chamber 3. When the layer of sterilizing medium has been applied to the web 1 in the chamber 3, the web emerges from the chamber 3, and after advancing a prearranged distance enters the chamber 4 where the sterilizing agent is removed from the web. The distance between the two chambers 3 and 4 (having regard to the speed of advance of the web) determines the time during which the sterilizing agent is permitted to act upon the web, and the distance between the chambers is appropriately selected so that the time is sufficient to ensure the desired bactericidal effect. The sterilizing agent is removed from the web in Chamber 4 by stream of hot air which is generated in a water ring compressor 24. The water ring compressor is of known type and the compressed air generated is passed through a water separator 25 which separates residual sealing water from the air, and a heating element 26 to the chamber 4. After a prearranged period during which the sterilizing agent is evaporated and removed from the chamber, the air mixed with the sterilizing agent is returned to the water ring compressor 24 where it is relieved of sterilizing agent mixed with it and is separated in the water separator 25.

It is important for the method in accordance with the invention that the air used can absorb a prearranged amount of hydrogen peroxide. To ensure this and to avoid the hydrogen peroxide being mixed with the moisture normally present in the air, dehydrated air is used. This is obtained by compressing the air in the compressor 11 to a pressure of 6—7 kg/cm². The air heated by the compression is then passed to a collar 12 in which it is cooled to a temperature of 15—20°C. By this means a large portion of the moisture carried by the air is precipitated and is removed in the following moisture trap 13. The resulting dehydrated air now has an absolute moisture content reduced from approximately 0.01 kg/kg to 0.003 kg/kg.

The dehydrated air is passed partly through the heating element 17 to the mixing chamber 18, and partly to the ultrasonic nozzle 19 to operate it and generate the standing wave which atomizes the sterilizing agent fed to the ultrasonic nozzle. The standing wave has a frequency of approximately 30 kHz and breaks down the sterilizing agent pumped-in to a mist with a droplet size of approximately 10 micrometre. The mist is sprayed directly into the mixing chamber 18 where it encounters and is mixed with the bulk of the dehydrated air from the moisture trap 13, which in the heating device 17 is heated to a high temperature. At this high temperature the droplets of sterilizing agent forming the injected mist directly come to boil and are vaporized. The temperature of the mixture is reduced thereby to approximately 100°C and the condensation temperature or dew-point at an absolute moisture of approximately 0.150 kg/kg will be approximately 60°C. This dew-point has been found to ensure condensation on the web 1 even under unfavourable conditions, e.g. at high temperature of the outside air. The dew-point may be varied upwards or downwards by a variation of the quantity of sterilising agent and the quantity of air in relation to one another.

The mixture of sterilizing agent vapour and air which reaches chamber 3 is guided in such a manner that is flows against the web 1 through the whole length of the chamber. During this flow, the part of the mixture which comes into contact with the web is cooled and the sterilizing agent condenses as a uniform layer on the surface of the web. In connection with the condensation heat is liberated which passes to the web and warms it. The condensation continues until the temperature difference is reduced (on the assumption that the material 1 has not already passed out of the chamber 3 before equality of temperature). Since the walls of chamber 3, at least to begin with, are at a lower temperature than the air mixture, the sterilizing agent also condenses on them. The excess sterilizing agent is removed from the chamber 3 through the pipe 23 and may be collected in a vessel. The air escaping from the chamber 3 still contains a certain amount of sterilizing agent, and the mixture may be passed to the water ring compressor 24, where the sterilizing agent is mixed with the sealing water and separated in the moisture trap 25.

After a suitable reaction period, the steri-

lizing agent is removed from the web 1, as described earlier, in the chamber 4, through which hot air is blown continuously at a temperature of approximately 130°C. It is appropriate to return the used air from this chamber also through a return line to the water ring compressor 24 for the recover of sterilizing agent.

The sterilizing agent may alternatively be removed from the web in any other suitable manner, e.g. by heating the web by a heating element of the infra red type.

When the web 1 has been wholly freed from sterilizing agent it advances, while being protected from bacteria and contaminations, through the packaging machine where, as described earlier, it is converted to a tube 6 which is filled with sterile contents, transversely sealed and cut into individual, completely filled, containers.

The method and the apparatus in accordance with the invention may be used on any of the known types of packaging machines. When the apparatus is to be used in association with a machine which operates with pre-formed blanks, the chambers 3 and 4 are modified in a suitable manner, and adapted to the shape of the blanks. The functional description described, however, applies to all types of packaging machines.

As a sterilizing agent preferably a mixture of 35% hydrogen peroxide and 65% water by weight is used, but other proportions and other suitable liquid sterilizing agents may be used. The numerical values specified in the description relate to the case where hydrogen peroxide and water are used.

When a sterile packaging machine is started up it is essential that the parts of the machine which will come into contact with the contacts or the web are presterilized. It is known to carry out this pre-sterilization in any of a number of different manners. For the method and apparatus in accordance with the invention it is appropriate that, as during operation of the machine, the mixture of air and sterilizing agent generated in the mixing chamber 18 should be used. When the machine is to be pre-sterilized, the outlet of the mixing chamber 18 is connected in such a manner to the parts of the machine which are to be pre-sterilized (i.e. the supply pipe for contents located in the tubular part of the web, the elements for the regulation of the liquid contents level, the chamber in which the web 1 runs from the chamber 3 to the sealing station etc.), that a closed system is created through which the mixture can circulate. Since the walls of the pipes and ducts through which the mixture circulates have a temperature lower than the dew-point of the mixture, a condensation of the sterilizing agent takes place. The mixture is caused to flow through the machines for a sufficiently long time to ensure that the sterilizing agent is condensed on all the surfaces which are to be sterilized. When the feed of sterilizing agent mixture has ceased, the condensed sterilizing agent is removed, after a suitable reaction time, by causing hot air to flow through the ducts. By this means, the walls are heated and the sterilizing agent vaporized which then accompanies the hot air out of the machine. The inflow of hot air is achieved by connecting the water ring compressor 24, the water separator 25 and the heating element 26 through a suitable valve to the sterilized spaces. In this manner pre-sterilization of the machine is effected by use of the method and apparatus in accordance with the invention and without major modification. The pre-sterilization is effective, since, similarly to what happens in the sterilization of the web, the sterilizing agent condenses on each component and in each space along the path followed by the web, provided only that the said components or spaces are at a sufficiently low temperature for condensation.

Although the method in accordance with the invention is intended primarily to be used in packaging machines, and has been described in connection therewith, the same principle can be used for any article whatever, provided that the same is introduced into a mixture of sterilizing vapour and water vapour at an appropriate temperature.

WHAT WE CLAIM IS:—

1. A method of sterilising the surface of an article or material wherein the surface at a relatively low temperature is exposed in a substantially closed chamber to an atmosphere comprising a vaporised liquid sterilising agent and a gas mixed in another chamber remote from, and conducted and delivered into, said first-mentioned chamber under conditions which ensure condensation of some of said vapour on said surface, and, after a pre-arranged period of time, said condensed sterilising agent is removed from said surface.

2. A method as claimed in Claim 1 wherein the said vaporised sterilizing agent is mixed with a stream of hot air.

3. A method as claimed in Claim 2 wherein the hot air is dehydrated before mixing with said vapour.

4. A method as claimed in Claim 1, 2 or 3 wherein the sterilizing agent is atomised to particles of size substantially within the range 5—15 micrometre.

5. A method as claimed in Claim 2 or in Claim 3 or 4 when appendant to Claim 2 wherein the air mixed with said vaporised agent has a temperature within the range of 90°—120°C., and preferably 100°C.

6. A method as claimed in Claim 3, or in Claim 4 or 5 when appendant to Claim 3 wherein the dried air has a moisture con-

tent within the range 0.1—0.2 kg/kg, and preferably 0.15 kg/kg.

7. A method as claimed in any preceding claim wherein the sterilizing agent is hydrogen peroxide.

8. Apparatus adapted to carry into effect a method as claimed in any of Claims 1—7 comprising a chamber for preparing a mixture of vaporised liquid sterilizing agent and a gas, means for conducting and delivering said mixture to another chamber housing an article or material for sterilization under conditions which ensure condensation of some of said vapour on the surface thereof, and means for subsequently removing said condensate sterilizing agent from said surface and conducting it from said second-mentioned chamber.

9. Apparatus as claimed in Claim 8 comprising means for mixing hot air with said vaporized agent.

10. Apparatus as claimed in Claim 9 comprising means for dehydrating said air in the course of providing it for mixture with said vaporised agent.

11. Apparatus as claimed in Claim 10 wherein said dehydrating means comprise an air compressor, a cooler and a water separator.

12. Apparatus as claimed in any of Claims 8—11 wherein said means for vaporising the agent comprise an atomiser.

13. Apparatus as claimed in Claim 12 wherein said atomiser comprises an ultrasonic nozzle.

14. Apparatus as claimed in Claim 13 wherein said ultrasonic nozzle is actuated by compressed air.

15. Apparatus as claimed in any of Claims 8—14 comprising a chamber to which said vaporised agent is supplied, and means for passing an article or material through said chamber in order to expose the surface of the article or material to the vapour therein.

16. Apparatus as claimed in Claim 15 comprising a second chamber in which condensate is removed from the article or material, and means for passing the article or material through the second chamber.

17. Apparatus as claimed in Claim 16 comprising means to pass heated air through said second chamber in order to remove said condensate.

18. Apparatus as claimed in Claim 16 or 17 wherein said two chambers are spaced from one another to ensure a reaction period for the sterilizing condensate on the article or material in the course of passing successively through the two chambers.

19. A method of sterilizing an article or material substantially as described herein with reference to the accompanying drawing.

20. Apparatus for sterilizing an article or material substantially as described herein with reference to the accompanying drawing.

21. An article or material, and more especially packaging material, whenever sterilized in accordance with a method, or by use of apparatus, substantially as described herein with reference to the accompanying drawing.

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